

PATENT SPECIFICATION

793,792



Date of filing Complete Specification : Jan. 6, 1956.

Application Date : Feb. 1, 1955. No. 3013/55.

Complete Specification Published : April 23, 1958.

Index at Acceptance :—Classes 40(3), A5(D1 : R1 : S1); and 46, B10AX.

International Classification :—C02b. G08c.

COMPLETE SPECIFICATION.

Improvements in or relating to the Determination of Impurities in Water.

We, CENTRAL ELECTRICITY AUTHORITY (formerly BRITISH ELECTRICITY AUTHORITY), a British Body Corporate, of Central (formerly British) Electricity House, Trafalgar Buildings, London, S.W.1, and HAROLD FREDERICK JAMES SCRASE, a British Subject, of 18 The Crescent, Southwick, Sussex, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement :—

This invention is for improvements in or relating to the determination of impurities in water and is particularly concerned with the quantitative determination of silica in water supplies.

In industrial plants requiring water of extremely high purity at all times e.g. feed water for high-pressure boiler plants, the water is taken from water treatment installations which include one or more ion-exchange beds for the purpose of removing silica from the water. Such ion-exchange beds periodically become exhausted and must be regenerated before they are of further use.

It is an object of the present invention to provide a method and apparatus for the quantitative determination e.g. of the amount of silica present in a water supply and hence an indication of exhaustion and need for regeneration of a bed of ion-exchange material installed for the purpose of removing silica. The method and apparatus of the present invention may also be employed to determine other impurities in water, e.g. the presence of phosphate ions.

According to the present invention there is provided a method for the quantitative determination periodically of a non-gaseous impurity in a water supply which method comprises adding to successive samples of the

water at predetermined time intervals measured quantities of two or more reagents nephelometrically and/or colorimetrically responsive to said impurity, allowing an interval for opacity or colour development to take place and then passing the treated sample to an optical cell through which a beam of light passes on to a photo-electric cell, the response in the photo-electric cell being a measure of the impurity content of the sample.

In a preferred form of the present invention the amount of silica present in a water supply is determined by the use of a reagent responsive to silica consisting of an acid solution of ammonium molybdate used in conjunction with a solution of 1-amino-2-naphthol-4-sulphonic acid in sodium metabisulphite solution. A predetermined volume of the acid solution of ammonium molybdate is added to a predetermined volume of the water and, after a time lapse, a predetermined volume of the solution of 1-amino-2-naphthol-4-sulphonic acid in sodium metabisulphite solution is added, again followed by a time lapse of predetermined duration, arrangements being made during the whole of this period for the mixture to be adequately stirred. The silica in the water reacts with the molybdate solution, yielding a compound which reacts with the sulphonic acid solution and produces a blue colouration. The density of this blue colouration, which is measured photo-electrically, is a direct indication of the amount of silica in the water.

The present invention also includes apparatus for the quantitative determination of an impurity in a water supply, which apparatus comprises means for adding at predetermined time intervals measured quantities of two or more reagents nephelometrically and/or colorimetrically responsive to the impurity intermittently to successive samples of the

[Price 3s. 6d.]

water, means for agitating the mixture and means for passing the treated samples to an optical cell and for measuring photo-electrically the resultant change in colour and/or opacity of the mixture in the optical cell.

The means for measuring the resultant change in colour and/or opacity of the treated water sample may comprise a source of light and a photo-electric cell on opposite sides of the optical cell. The source of light is of predetermined intensity and range of wavelength.

Where the characteristics of the water and/or photo-electric cell are liable to vary it is desirable to divide the water sample, one portion being treated in the manner indicated above, the other portion not being treated, these two portions then being fed to two separate identical optical cells, the light being transmitted through the two optical cells and impinging respectively on to two similar photo-electric cells, connected to an apparatus responding only to the differential variations of the electric current generated in the cells.

The apparatus preferably includes an arrangement whereby the temperature of the respective components is maintained steady and the whole apparatus is desirably encased such that freedom from atmospheric dust settlement is ensured.

When the present invention is used to determine the amount of silica present in a water supply taken from a water treatment installation which includes one or more beds of anion-exchange material for the removal of silica, the response in the photo-electric cell or cells may be employed to initiate the operation of regeneration of the anion-exchange material when more than a predetermined amount of silica is present in the water.

Thus, the invention further includes a water treatment installation including an anion-exchange bed for the removal of silica from the water and means for regenerating the anion-exchange material where necessary, in which installation an apparatus as hereinbefore described is connected to the effluent pipe from the anion-exchange bed to determine the amount of silica in the effluent, the photo-electric cell or cells of said apparatus indicating the amount of silica present in the effluent and/or initiating the operation of regeneration of the anion-exchange bed when more than the predetermined amount of silica is present.

Following is a description by way of example and with reference to the accompanying drawings of two embodiments of the present invention. The single Figure of the drawing filed with the Provisional Specification is a purely diagrammatic representation of the apparatus. The single Figure of the drawing filed with the Complete Specification

is a diagrammatic representation of an alternative apparatus.

The water under test is obtained from a sampling point which is located in the discharge pipe of an anion-exchange bed installed in a water treatment installation for the purpose of removing silica from feed water for high-pressure boiler plants.

Referring to the drawing filed with the Provisional Specification water flowing through the sampling tube is admitted into a constant head container 11, the water level being maintained by means of an overflow pipe. The constant head container supplies water to a sample dispenser 12 via an electrically operated solenoid valve 13 which opens at a predetermined time interval. When valve 13 shuts and the electrically operated solenoid valve 14 opens a measured volume of the water under test is delivered to the reaction vessel 15. At predetermined time intervals valves 16 and 17, which are also electrically operated, open to discharge to the reaction vessel 15 measured volumes of reagents responsive to silica from two reagent dispensers. The reagent dispensers each comprise a main reservoir 18 and 19, a flow rate controller 20 and 21, an overflow collector 22 and 23 and a lower reservoir 24 and 25. Facility is provided to return the contents of the lower reservoirs 24 and 25 to the respective main reservoirs 18 and 19 by means of a blow-bulb. Master cocks 26 and 27 may be provided which are electromagnetically controlled to operate at set time intervals. The flow through controllers 20 and 21 is arranged that it is too small to affect the accuracy of the dispensing operation and does not need to be shut off when valves 16 and 17 open.

The reaction vessel 15 which receives the water sample and the measured volumes of reagents is provided with a stirrer 28 driven by a synchronous electric motor of speed one revolution per second. The outlet of the reaction vessel 15 is connected to a measuring cell 29, the outlet of which is controlled by an electrically operated solenoid valve 30 which, on opening, allows displacement of the mixture present in the cell by that in the reaction vessel. The cell volume, being preferably smaller than that of the mixture in the reaction vessel, ensures complete replacement in the cell. The cell arrangement comprises a light source 31 supplied from a constant voltage transformer. This light passes through a red filter 32 and a variable aperture 33 before passing through the cell 29 and impinging upon the photo-electric cell 34 which is connected to a microammeter (not shown). Thus the photo-electric cell will respond to and the microammeter will operate in accordance with changes in intensity of the light impinging upon the

photo-electric cell due to changes in the transmission of light through the cell 29.

Since the light which can be transmitted will vary with variation in the colour or opacity of the treated sample and thus with the silica content of the original sample, it will be seen that the indication on the microammeter will be proportional to the amount of silica in the sample.

In a modified arrangement designed to avoid errors in the reading given by the microammeter which might occur due to changes in the transmissibility of the treated sample resulting from changes in the transmissibility of the original water, or due to functional changes in the property of the photo-cell, a supply of water is taken direct from the constant head and passed continuously through a duplicated cell arrangement with a second photo-electric cell. This cell is connected electrically with the first and connected to electronic apparatus which indicates only the differential between the two cells.

Provision is made via cock 35 to flush the cell system with untreated water in order to verify the zero accuracy of the apparatus.

The whole apparatus is encased in a dust-proof casing, and is controlled thermostatically within predetermined temperature limits. Portions of the apparatus are necessarily constructed in silica free material, e.g. polythene.

Referring to the drawing filed with the Complete Specification, the apparatus comprises four liquid dispensers 36, 37, 38 and 39, a reaction vessel 40 and a measuring cell 41. The sample dispenser 36 is supplied with water via a constant head container 42 which is made to deliver 50 mls. water when required by two electrically operated solenoid valves 43 and 44. Valve 43 opens and allows the dispenser 36 to fill, after which it closes and valve 44 opens. This discharges the measured volume to the reaction vessel 40.

The reagent dispensers 37, 38 and 39 are identical and comprise polythene reservoirs fitted with Mariotte tubes 45 feeding dispensing pipettes 46 of polythene via electrically operated solenoid valves 47, 48 and 49. Further electrically operated solenoid valves 50, 51 and 52 discharge the measured volume of the reagents to the reaction vessel 40 as required. The actual volume of reagent dispensed is determined by adjusting the level of the Mariotte tube with respect to the vertical pipette tube.

The reaction vessel 40, which is also of polythene, receives all the outlets from the dispensers and is equipped with a polyethylene stirrer 53 driven by a synchronous motor 54 of speed one revolution per second. The reaction vessel 40 is connected to a "Perspex" optical cell 41, which has a

further solenoid valve 55 at its outlet. The operation of this valve delivers the solution to the optical cell, displacing that already there from the previous sequence. The volume of the cell is considerably smaller than the sample volume, so that almost complete flushing is attained during one change-over.

A light source 56 consisting of a 12-volt 36 watt bulb with a concave focusing mirror is passed through a monochromatic spectrum filter 609; through the cell 41 containing the solution and directed onto a vacuum photo-electric cell 58, which with the appropriate electronic circuit, transmits the absorption obtained to a recording meter.

The solenoid valves are so constructed that all surfaces exposed to reagent solutions are either of ebonite or polythene, and are controlled in sequence by a time clock which operates the system as follows:—

Valve 43 opens and passes water under test to the dispenser 36, which is then discharged to the reaction vessel 40 as already described. Simultaneously, the first two reagent dispensers 38 and 39 discharge to the reaction vessel measured volumes of hydrochloric acid and ammonium molybdate solutions. The valve 49 of the third dispenser 37 opens during this period and allows 1-amino-2-naphthol-4-sulphonic acid solution to pass from the reservoir to the pipette. This measured volume is discharged to the reaction vessel after five minutes. A further 12 minutes reaction time is allowed before valve 55 operates and passes the solution to the optical cell for measurement. During this time the reagent dispensers containing the acid and ammonium molybdate are recharged in readiness for the next sequence by the operation of valves 47 and 48.

When not in use the reagent circuits of the time clock are switched out, but the water sequence continues, so that the meter is continuously flushed with fresh water and a zero record is obtained. Under these conditions the meter zero can be adjusted. The time required for the complete sequence is 20 minutes, which is sufficiently frequent for control purposes.

If circumstances require the prevention of phosphate interference in the reaction the meter is easily adaptable to take a further reagent dispenser for oxalic acid solution, which would be added in its correct sequence by the time clock.

What we claim is:—

1. A method for the quantitative determination periodically of a non-gaseous impurity in a water supply which method comprises adding to successive samples of the water at predetermined time intervals measured quantities of two or more reagents nephelometrically and/or colorimetrically responsive to said impurity, allowing an interval

for opacity or colour development to take place and then passing the treated sample to an optical cell through which a beam of light passes on to a photo-electric cell, the response in the photo-electric cell being a measure of the impurity content of the sample.

2. A method as claimed in Claim 1 wherein the amount of silica present in a water supply is determined by the use of a reagent responsive to silica consisting of an acid solution of ammonium molybdate used in conjunction with a solution of 1-amino-2-naphthol-4-sulphonic acid in sodium metabisulphite solution.

3. Apparatus for carrying out the quantitative determination of an impurity in a water supply by a method as claimed in Claim 1, which apparatus comprises means for adding at predetermined time intervals measured quantities of two or more reagents nephelometrically and/or colorimetrically responsive to the impurity intermittently to successive samples of the water, means for agitating the mixture and means for passing the treated samples to an optical cell and for measuring photo-electrically the resultant change in colour and/or opacity of the mixture in the optical cell.

4. Apparatus as claimed in Claim 3 wherein the means for measuring the resultant change in colour and/or opacity of the treated water sample comprise a source of light and a photo-electric cell on opposite sides of the optical cell.

5. Apparatus as claimed in Claim 3 or Claim 4 wherein electrically operated solenoid valves are employed to provide from the water supply successive samples of the water, to add to such samples at predetermined time intervals measured quantities of the reagents,

and to transfer the treated samples to the optical cell at predetermined time intervals.

6. A method as claimed in Claim 1 or Claim 2 wherein the water samples are divided, one portion being treated with a predetermined quantity of the reagents nephelometrically and/or colorimetrically responsive to said impurity the other portion not being treated, the two portions then being fed to two separate identical optical-cells, the light being transmitted through the two optical cells and impinging respectively on to two similar photo-electric cells, connected to an apparatus responding only to the differential variations of the electric current generated in the cells.

7. A water treatment installation including an anion-exchange bed for the removal of silica from the water and means for regenerating the anion-exchange material where necessary, in which installation an apparatus as claimed in any one of Claims 3, 4 or 5 is connected to the effluent pipe from the anion-exchange bed to determine the amount of silica in the effluent, the photo-electric cell or cells of said apparatus indicating the amount of silica present in the effluent and/or initiating the operation of regeneration of the anion-exchange bed when more than the predetermined amount of silica is present.

8. Apparatus for the quantitative determination of an impurity in a water supply substantially as described in the specific examples hereinbefore set forth with reference to the accompanying drawings.

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Chartered Patent Agents.

PROVISIONAL SPECIFICATION.

Improvements in or relating to the Determination of Impurities in Water.

We, BRITISH ELECTRICITY AUTHORITY, a British Body Corporate, of British Electricity House, Trafalgar Buildings, London, S.W.1, and HAROLD FREDERICK JAMES SCRASE, a British Subject, of 18 The Crescent, Southwick, Sussex, do hereby declare this invention to be described in the following statement:—

This invention is for improvements in or relating to the determination of impurities in water and is particularly concerned with the quantitative determination of silica in water supplies.

In industrial plants requiring water of extremely high purity at all times, e.g. feed water for high-pressure boiler plants, the water is taken from water treatment installations which include one or more ion-exchange

beds for the purpose of removing silica from the water. Such ion-exchange beds periodically become exhausted and must be regenerated before they are of further use.

It is an object of the present invention to provide a method and apparatus for the quantitative determination e.g. of the amount of silica present in a water supply and hence an indication of exhaustion and need for regeneration of a bed of ion-exchange material installed for the purpose of removing silica. The method and apparatus of the present invention may also be employed to determine other impurities in water, e.g. the presence of phosphate ions.

According to the present invention there is provided a method for the quantitative deter-

mination periodically of an impurity in a water supply which method comprises adding to successive samples of the water a predetermined quantity of a reagent or reagents
 5 nephelometrically and/or colorimetrically responsive to said impurity and passing a beam of light through the treated sample on to a photo-electric cell, the response in the photo-electric cell being a measure of the
 10 impurity content of the sample.

In a preferred form of the present invention the amount of silica present in a water supply is determined by the use of a reagent responsive to silica consisting of an acid solution of ammonium molybdate used in conjunction with a solution of 1-amino-2-naphthol-4-sulphonic acid in sodium metabisulphite solution. A predetermined volume of the acid solution of ammonium molybdate is added to a predetermined volume of the water and, after a time lapse, a predetermined volume of the solution of 1-amino-2-naphthol-4-sulphonic acid in sodium metabisulphite solution is added, again followed by a time
 20 lapse of predetermined duration, arrangements being made during the whole of this period for the mixture to be adequately stirred. The silica in the water reacts with the molybdate solution, yielding a compound which reacts with the sulphonic acid solution and produces a blue coloration. The density of this blue coloration, which is measured photo-electrically, is a direct indication of the amount of silica in the water.

The present invention also includes apparatus for the quantitative determination of an impurity in a water supply, which apparatus comprises means for adding predetermined quantities of a reagent or reagents nephelometrically and/or colorimetrically responsive to the impurity intermittently to successive samples of the water, means for agitating the mixture and means for measuring photo-electrically the resultant change in colour and/or opacity of the mixture.

The means for measuring the resultant change in colour and/or opacity of the treated water sample may comprise a transparent vessel having a source of light and a photo-electric cell on opposite sides thereof. The source of light is of predetermined intensity and range of wavelength.

Where the characteristics of the water and/or photo-electric cell are liable to vary it is desirable to divide the water sample, one portion being treated in the manner indicated above, the other portion not being treated, these two portions then being fed to an apparatus which responds only to the difference between the two portions. This may conveniently comprise an arrangement whereby the light transmitted through the two portions of the sample impinges respectively on to similar photo-electric cells, connected to an apparatus responding only to the

differential variations of the electric current generated in the cells.

The apparatus preferably includes an arrangement whereby the temperature of the respective components is maintained steady and the whole apparatus is desirably encased such that freedom from atmospheric dust settlement is ensured.

When the present invention is used to determine the amount of silica present in a water supply taken from a water treatment installation which includes one or more beds of anion-exchange material for the removal of silica, the response in the photo-electric cell or cells may be employed to initiate the operation of regeneration of the anion-exchange material when more than a predetermined amount of silica is present in the water.

Thus, the invention further includes a water treatment installation including an anion-exchange bed for the removal of silica from the water and means for regenerating the anion-exchange material where necessary, in which installation an apparatus as hereinbefore described is connected to the effluent pipe from the anion-exchange bed to determine the amount of silica in the effluent, the photo-electric cell or cells of said apparatus indicating the amount of silica present in the effluent and/or initiating the operation of regeneration of the anion-exchange bed when more than the predetermined amount of silica is present.

Following is a description by way of example and with reference to the accompanying drawing of one embodiment of the present invention. The single Figure of the drawing is a purely diagrammatic representation of the apparatus.

The water under test is obtained from a sampling point which is located in the discharge pipe of an anion-exchange bed installed in a water treatment installation for the purpose of removing silica from feed water for high-pressure boiler plants.

The water flowing through the sampling tube is admitted into a constant head container 11, the water level being maintained by means of an overflow pipe. The constant head container supplies water to a sample dispenser 12 via an electrically operated solenoid valve 13 which opens at a predetermined time interval. When valve 13 shuts and the electrically operated solenoid valve 14 opens a measured volume of the water under test is delivered to the reaction vessel 15. At predetermined time intervals valves 16 and 17, which are also electrically operated, open to discharge to the reaction vessel 15 measured volumes of reagents responsive to silica from two reagent dispensers. The reagent dispensers each comprise a main reservoir 18 and 19, a flow rate controller 20 and 21, an overflow collector 22 and 23 and a lower

reservoir 24 and 25. Facility is provided to return the contents of the lower reservoirs 24 and 25 to the respective main reservoirs 18 and 19 by means of a blow-bulb. Master cocks 26 and 27 may be provided which are electromagnetically controlled to operate at set time intervals. The flow through controllers 20 and 21 is arranged that it is too small to affect the accuracy of the dispensing operation and does not need to be shut off when valves 16 and 17 open.

The reaction vessel 15 which receives the water sample and the measured volumes of reagents is provided with a stirrer 28 driven by a synchronous electric motor of speed one revolution per second. The outlet of the reaction vessel 15 is connected to a measuring cell 29, the outlet of which is controlled by an electrically operated solenoid valve 30 which, on opening, allows displacement of the mixture present in the cell by that in the reaction vessel. The cell volume, being preferably smaller than that of the mixture in the reaction vessel, ensures complete replacement in the cell. The cell arrangement comprises a light source 31 supplied from a constant voltage transformer. This light passes through a red filter 32 and a variable aperture 33 before passing through the cell 29 and impinging upon the photo-electric cell 34 which is connected to a microammeter. Thus the photo-electric cell will respond to and the microammeter will operate in accordance with changes in intensity of the light impinging upon the photo-electric cell due to changes in the transmission of light through the cell 29.

Since the light which can be transmitted will vary with variation in the colour or

opacity of the treated sample and thus with the silica content of the original sample, it will be seen that the indication on the microammeter will be proportional to the amount of silica in the sample.

In a modified arrangement designed to avoid errors in the reading given by the microammeter which might occur due to changes in the transmissibility of the treated sample resulting from changes in the transmissibility of the original water, or due to functional changes in the property of the photo-cell, a supply of water is taken direct from the constant head and passed continuously through a duplicated cell arrangement with a second photo-electric cell. This cell is connected electrically with the first and connected to electronic apparatus which indicates only the differential between the two cells.

Provision is made via cock 35 to flush the cell system with untreated water in order to verify the zero accuracy of the apparatus.

The whole apparatus is encased in a dust-proof casing, and is controlled thermostatically within predetermined temperature limits. Portions of the apparatus are necessarily constructed in silica free material, e.g. polythene.

If desired a recording device may be installed in place of the microammeter.

It will be understood that a number of dispensers may be incorporated in the apparatus to operate in a predetermined sequence.

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Abingdon : Printed for Her Majesty's Stationery Office, by Burgess & Son (Abingdon), Ltd.—1958.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2,
from which copies may be obtained.

793,792 COMPLETE SPECIFICATION

1 SHEET

This drawing is a reproduction of the Original on a reduced scale.

